Beryllium Measurement in Commercially Available Wet Wipes

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- Mention of commercial products in this presentation does not imply endorsement by the author, SRNS, SRS, or the U.S. Department of Energy (DOE).

- The speaker is an employee of a Department of Energy (DOE) contractor, and is not a spokesperson for DOE itself.

History

- Beryllium sample increase is expected from work generated by American Resource and Recovery Act
- Current Analysis (HF acid digestion and Column Separation, measurement by sequential ICP-ES) is very labor intensive, resulting in long turnaround times.
- No funds are available for additional laboratory personnel or more expensive equipment
- Need emerged for an inexpensive and less labor intensive beryllium measuring method
- Optical Fluorescence Method is less labor intensive
- Costs are significantly less than to purchase an ICP-MS

Challenges

- Savannah River Site field IH prefer individually packaged pre-wetted wipes to perform beryllium sampling
- The ASTM lead wipe standard requires that sample media be wet and individually packaged. A similar beryllium wipe standard is expected to be established in the near future.
- Most customers currently using the optical fluorescence method use dry MCE filters
- More ammonium bifluoride is required to submerge the commercially available wipes into the solution (detection limit challenges)

Palintest™, Ghostwipe™, and MCE Blank Testing

	Palintest™ wipe	Ghostwipe™	High Purity™ MCE filter
Blank: 0 µg Be	0.06µg	<0.005µg	<0.005µg
	0.03µg	<0.005µg	<0.005µg
	0.04µg		<0.005µg
			<0.005µg
Spiked with Be acetate	0.061µg (153%)	0.04µg (100%)	0.0038µg (93%)
Wipes: 0.04µg Be	0.057µg (139%)	0.041µg (103%)	0.0038µg (93%)
*Filters: 0.004µg	0.064µg (160%)	0.04µg (100%)	0.0038µg (93%)
	0.049µg (120%)	0.037µg (93%)	0.004µg (100%)
	0.063µg (154%)	0.038µg (93%)	0.004µg (100%)

* Filters were spiked with a concentration of beryllium that is less than the established method reporting limit.

Method Reporting Limit for MCE Filters

Standard Conc. Used =	0.08	0.08	0.08
Units	ppb	ppb	ppb
Other Parameters/Information:	filter	filter	filter
Replicate Number [1,2]	Measured Values	Measured Values	Measured Values
1	*0.070*	*0.070*	*0.050*
2	*0.070*	*0.070*	*0.070*
3	0.080	0.080	0.070
4	0.080	0.070	0.080
5	0.080	0.070	0.040
6	0.090	0.070	0.050
7	0.080	0.080	0.060
8	0.070	0.080	0.070
9	0.090	0.070	0.080
10	0.080	0.080	0.060
[1,2]Number Measurements (n)=	10	10	10
Standard Deviation (S)=	0.007	0.005	0.013
[4] Standard Conc. / S =	11	15	6
[5]Student's t (σ = 0.99, n-1) =	2.8214	2.8214	2.8214
[6]Lower Limit of Detection (LLD) =	0.02082	0.01457	0.03774
[6]Lower Limit of Quantitation (LLQ) =	0.10409	0.07285	0.18868
Lower Limit of Quantitation (µg) =	**0.002** ug/filter	**0.002** ug/filter	**0.004** ug/filter

Method Reporting Limit for Ghostwipes™

Standard Conc. Used =	0.10	0.10	0.10
Units	ppb	ppb	ppb
Other Parameters/Information:	ghostwipe	ghostwipe	ghostwipes
Replicate Number [1,2]	Measured Values	Measured Values	Measured Values
1	0.090	*0.090*	*0.100*
2	0.100	*0.090*	*0.100*
3	0.100	0.090	0.100
4	0.100	0.100	0.080
5	0.100	0.100	0.100
6	0.090	0.130	0.100
7	0.110	0.090	0.100
8	0.100	0.100	0.090
9	0.100		0.090
10	0.110		0.100
[1,2]Number Measurements (n)=	10	8	10
Standard Deviation (S)=	0.007	0.014	0.007
[4] Standard Conc. / S =	15	7	14
[5]Student's t (σ = 0.99, n-1) =	2.8214	2.9979	2.8214
[6]Lower Limit of Detection (LLD) =	0.01881	0.04066	0.01973
[6]Lower Limit of Quantitation (LLQ) =	0.09405	0.20329	0.09864
[7]Lower Limit of Quantitation (µg) =	**0.009**ug/wipe	**0.020** ug/wipe	**0.010** ug/wipe

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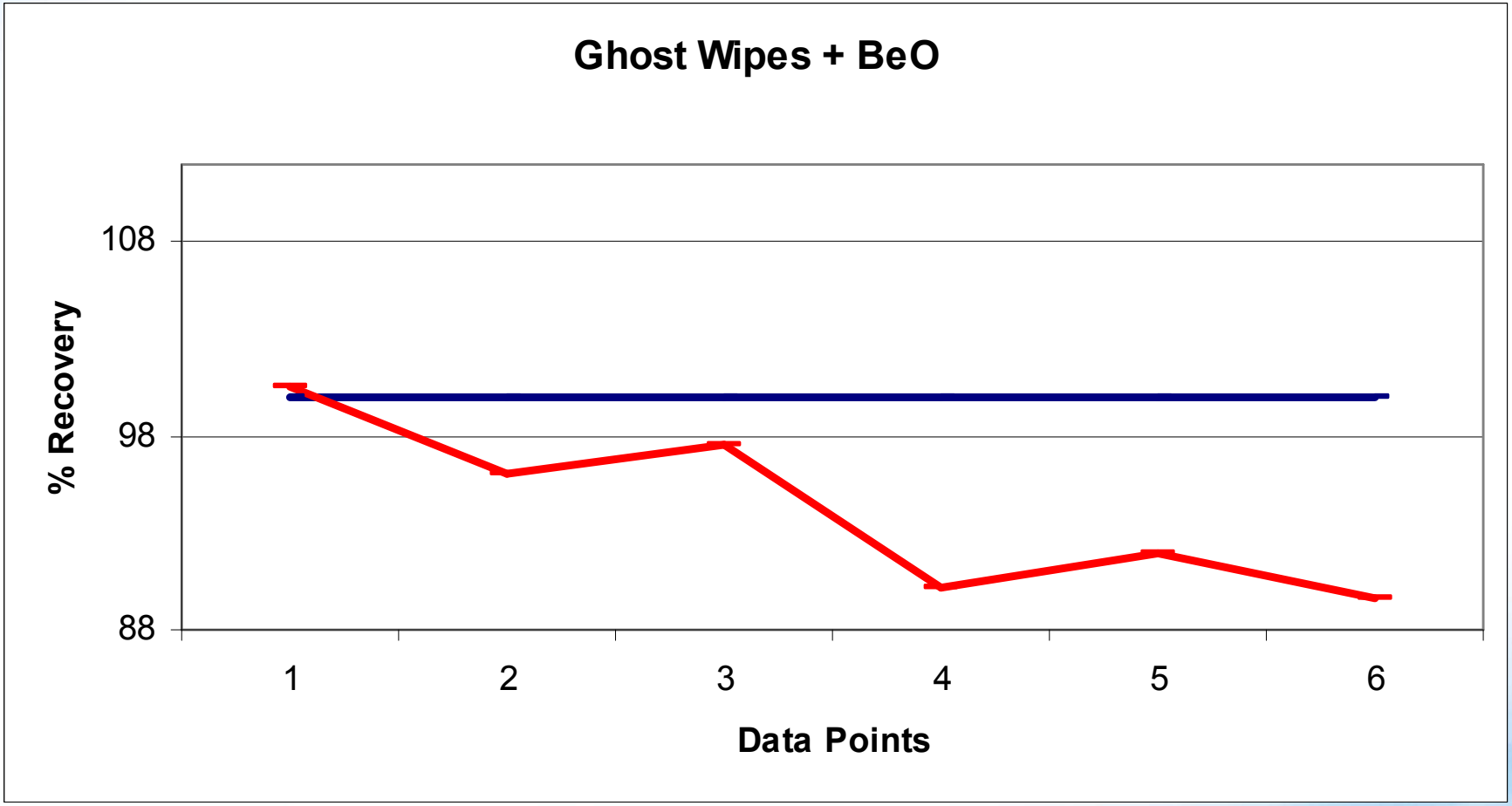
Recovery of Ghostwipes™ + HP™ MCE BeO Standards



Recovery of Ghostwipes™ + HP™ MCE BeO Standards

Concentration	Expected Value µg	Result ppb	Result µg	% Recovery
BeO 0.01*	0.01	0.14	0.014	140.0
BeO 0.01*	0.01	0.11	0.011	110.0
BeO 0.2	0.2	2.01	0.201	100.5
BeO 0.2	0.2	1.92	0.192	96.0
BeO 0.5	0.5	4.88	0.488	97.6
BeO 0.5	0.5	4.51	0.451	90.2
BeO 1.0	1	9.19	0.919	91.9
BeO 1.0	1	8.97	0.897	89.7

*Concentration below established Method Reporting Limit.

Interference Testing, Fe/Ti/Pu in Ghostwipes™

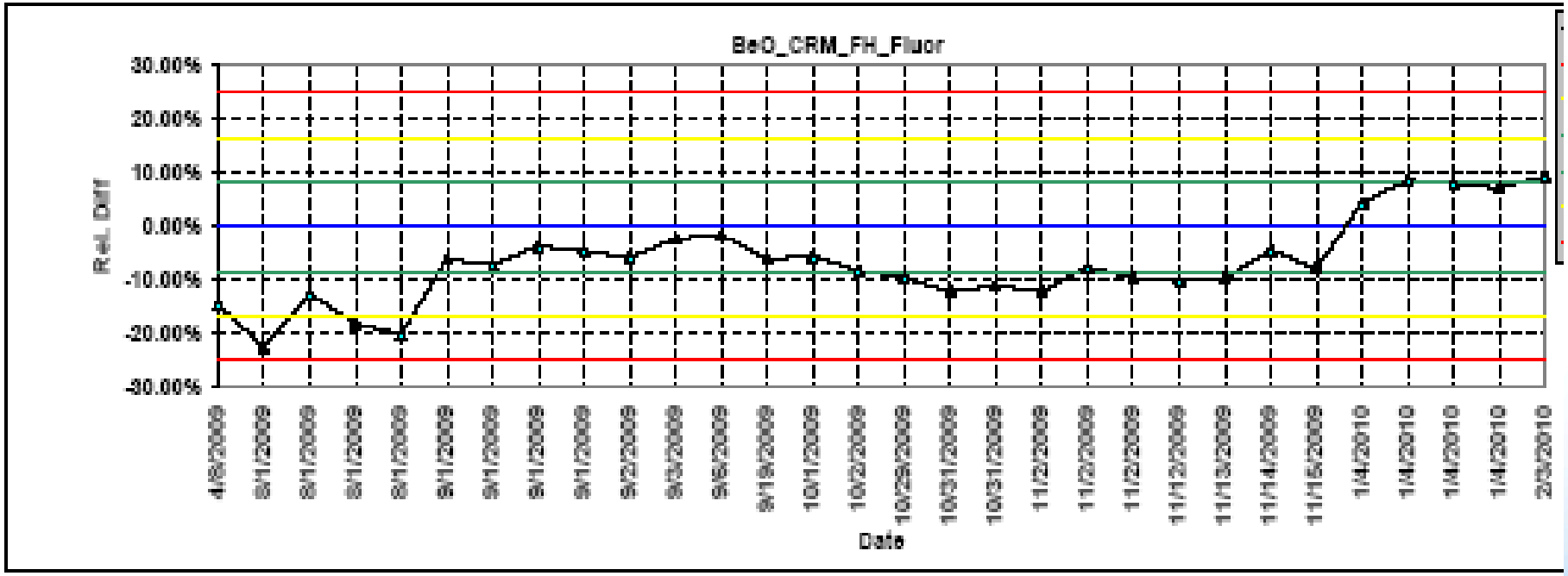
Interference	Conc Interference	Conc Be	Be result	Recovery	Interference	Conc Interference	Conc Be	Be result	Recovery
Fe	10 mg	0.2	0.2	**100**	Ti	2 mg	0.2	0.21	**105**
	10 mg	0.2	0.16	**125**		2 mg	0.2	0.22	**110**
	10 mg	2	1.74	**87**		2 mg	2	1.72	**86**
	10 mg	2	1.76	**88**		2 mg	2	1.69	**85**
	10 mg	0.2	0.21	**95**	Pu	~5E6dpm	0.2	0.21	**105**
	10 mg	0.2	0.2	**100**		~5E6dpm	0.2	0.2	**100**
	10 mg	0.2	0.27	**135**		~5E6dpm	2	1.85	**93**
	10 mg	0.2	0.23	**115**	HP-CRMD	n/a	0.1	0.104	**104**
	10 mg	2	1.84	**92**	HP-CRME	n/a	0.2	0.201	**101**
	10 mg	2	1.8	**90**					
Blank	n/a	0	0.04	n/a					
Blank	n/a	0.01	0.04	n/a					
Blank	n/a	0.01	0.02	n/a					

Ghostwipe™ Lot Comparison:

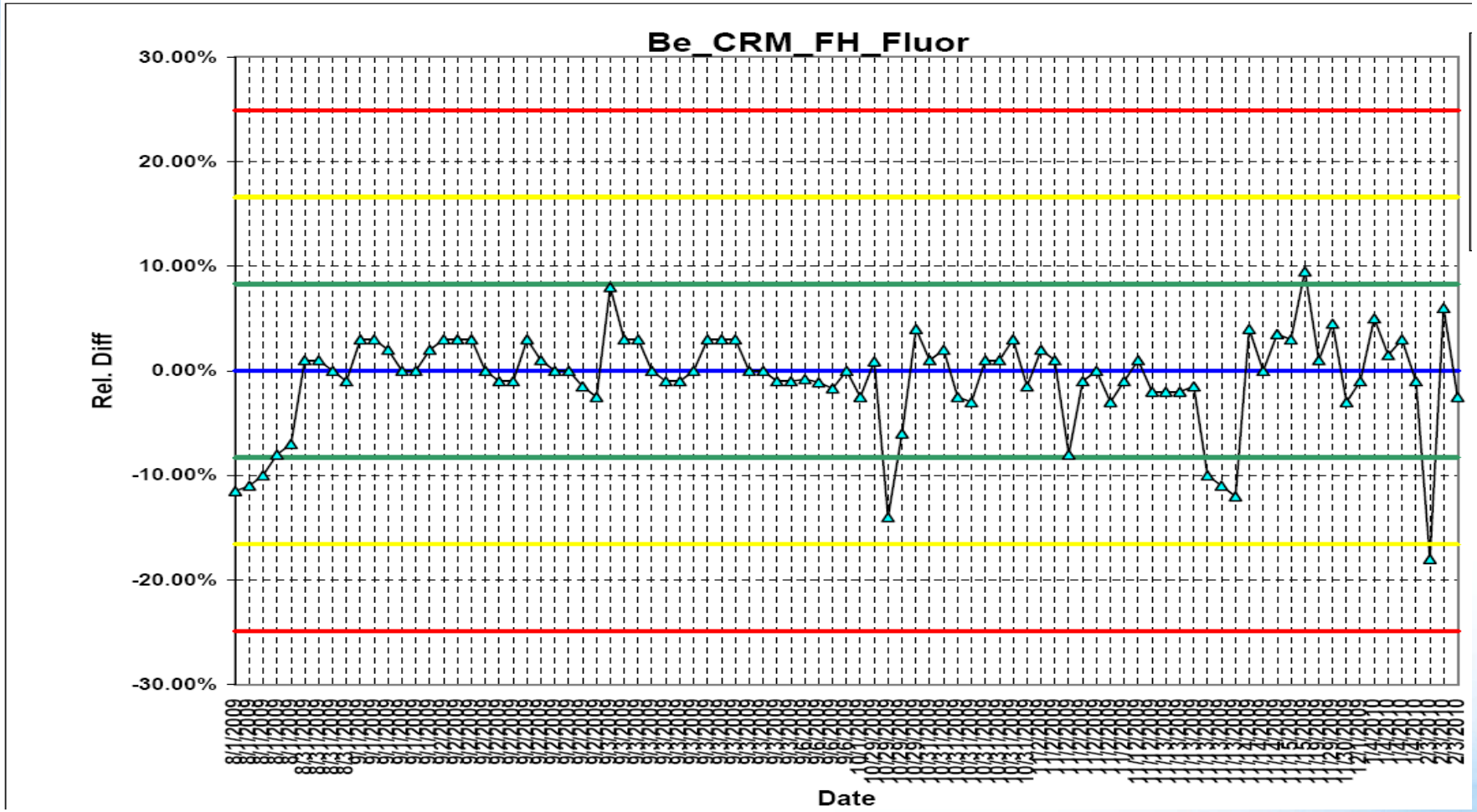
Ghost Wipe Lot Comparison			
	Lot 1	**Lot 2**	**Lot 3**
	<0.005µg	<0.005µg	<0.005µg
	<0.005µg	<0.005µg	<0.005µg

General Data, Beryllium by Optical Fluorescence

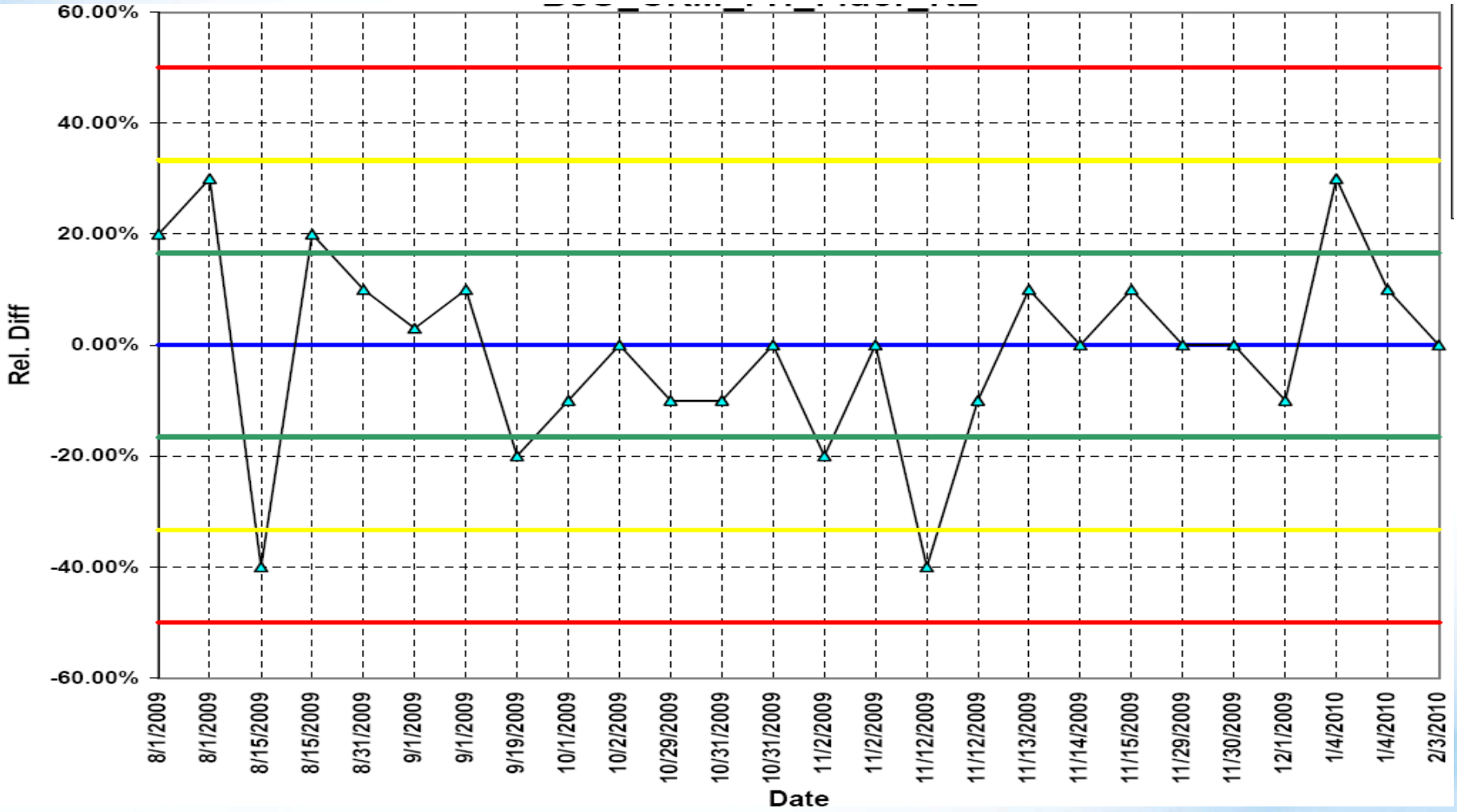
Quality Control Chart of Beryllium Oxide by Optical Fluorescence



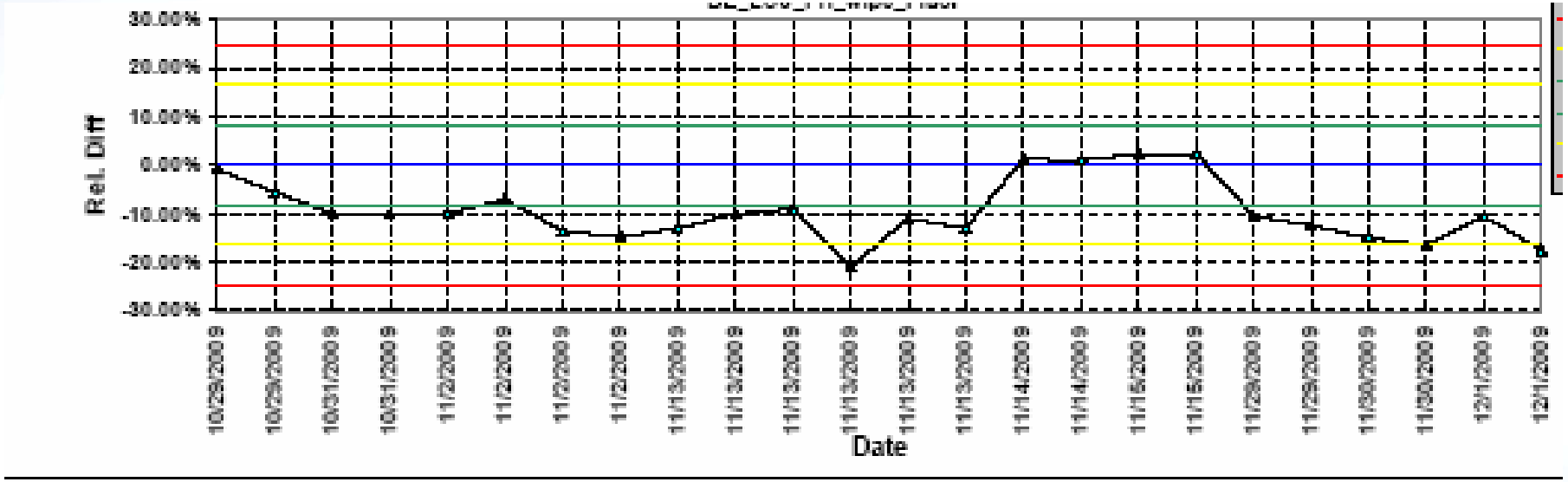
High Purity™ Certified Reference Material Beryllium Acetate Filters



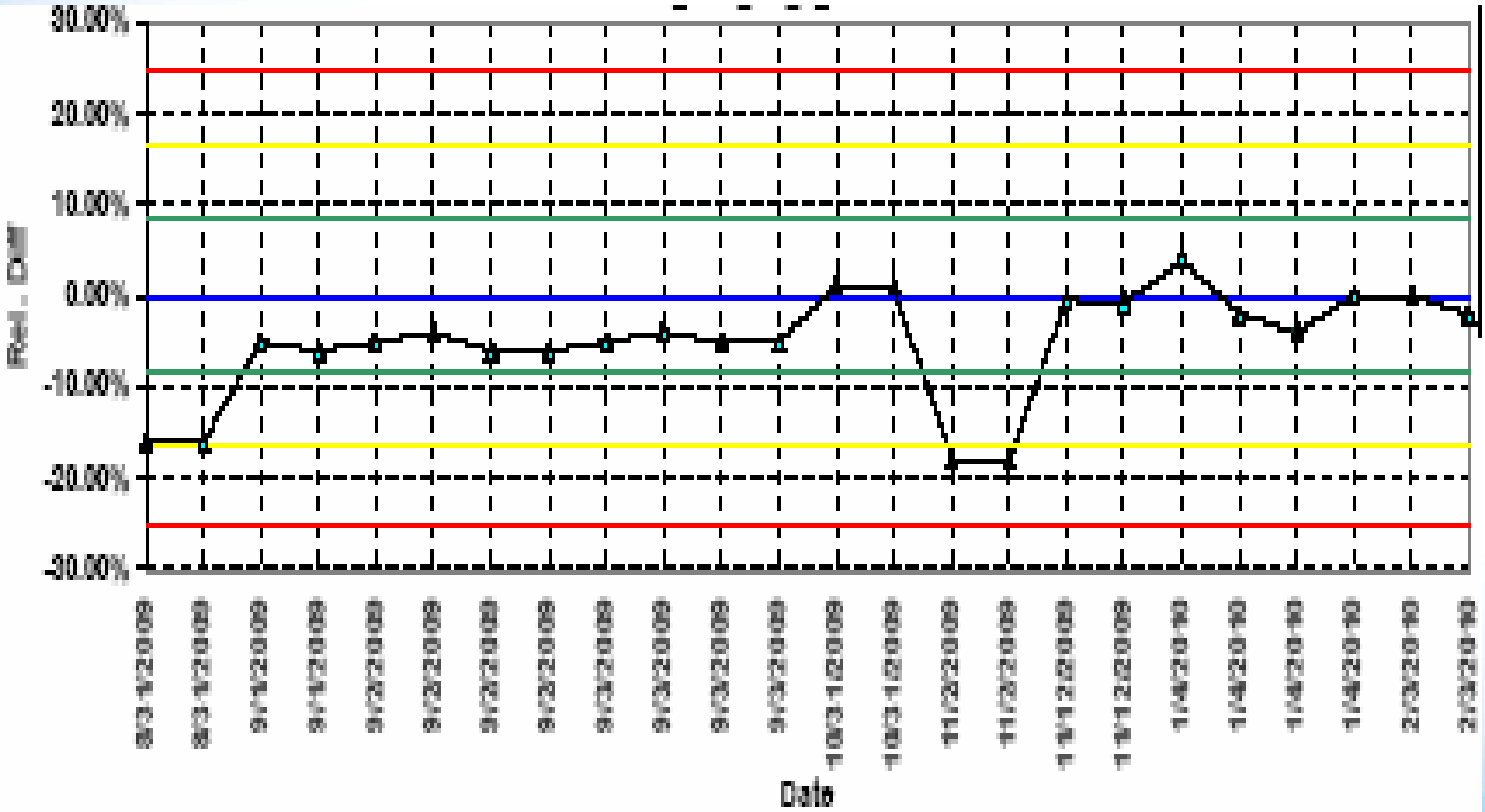
HP Beryllium Oxide Filters @ 0.01µg Be



Ghostwipes™ Spiked with Beryllium Acetate



High Purity™ Filters Spiked with Beryllium Acetate



Summary

- Savannah River Site currently uses Ghostwipes for all wet surface sampling. For Industrial Hygiene to convert to the optical fluorescence method, very little change would be required in the field.
- The addition of Optical Fluorescence as a Field of Testing allows the customer an option that should result in faster turnaround times and save money (labor dollars). The exact savings have not yet been calculated but are expected to be at least 50% of the current costs.
- The manual system is sufficient to handle SRNS current sample load, but if that changes, the option for an automated unit is available.

Future Needs

- There is no established standard for wet wipes used for beryllium surface sampling.
- Commercially available wipes are not required to adhere to a standard that ensures their suitability for beryllium measurement.
- Differences between lots in some brands of wipes is significant.
- There is no promise from any commercial provider that the wipes we use for beryllium sampling will not vary in material, preservative, or even background beryllium concentration, and this is a concern for ICP as well as Optical Fluorescence use.

Further Information

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